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MICROSTRUCTURAL DESIGN AND DEVELOPMENT OF PATH A PRIME-CANDIDATE ALLOY*

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SUMMARY

This work summarizes the results of microstructural design and alloy development in the Path A Prime Candidate Alloy (PCA). The basis for the microstructural design, basically, is the well-established MC-helium trapping mechanism and its correlation with improved swelling and embrittlement resistance in HFIR-irradiated 316 + Ti. A range of preirradiation microstructures were designed with variable combinations of matrix and grain-boundary precipitation prior to actual investigation of the PCA. The microstructural condition of the PCA as received from the vendor, however, was severely stringered with MC and inhomogeneous, making the material unfit for further microstructural investigation or manipulation. A fabrication investigation that correlated treatment with microstructure revealed that both initial homogenization followed by subsequent homogenization treatments during fabrication were most important for obtaining proper specimen material. Several problems were encountered with nitride formation and grain-size control, but those were solved to actually produce homogeneous specimen material with a uniform and completely variable and controllable grain size. The time-temperature-precipitation (TTP) behavior of the material after various mechanical treatments (0, 10 and 25% CW) was investigated to reveal the microstructures available and the thermal-mechanical treatments required to produce them. These were combined to produce preirradiation microstructures that had either fine or coarse MC distributions in the matrix or at the grain boundary in various combinations. These complex microstructures were produced in addition to the simple solution annealed or cold-worked microstructures.

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Alloy Chemistries (wt %)

	316	(316 + Ti)	Path A PCA
Cr	18.0	17.0	14.0
Ni	13.0	12.0	16.2
Mo	2.6	2.5	2.3
Mn	1.9	0.5	1.8
Si	0.8	0.4	0.4
Ti	0.05	0.23	0.24
C	0.05	0.06	0.05
P	0.013	0.01	0.01
S	0.016	0.013	0.003
N	0.005	0.006	0.01
B	0.0005	0.0007	0.0005
V	0.01	0.01	b
Nb	0.0005	0.01	b

[a]Balance iron + trace impurities.

[b]Not detectable.

I. MICROSTRUCTURAL DESIGN

BASED ON PRINCIPLES FROM:

- MECHANISM OF HELIUM-MC INTERFACIAL TRAPPING
- PROPERTIES-MICROSTRUCTURE CHARACTERIZATION AND CORRELATION FOR HFIR IRRADIATED 316 AND 316 + Ti
- NATURE OF PRECIPITATE PHASE DEVELOPMENT (IRRADIATION AND THERMAL AGING)

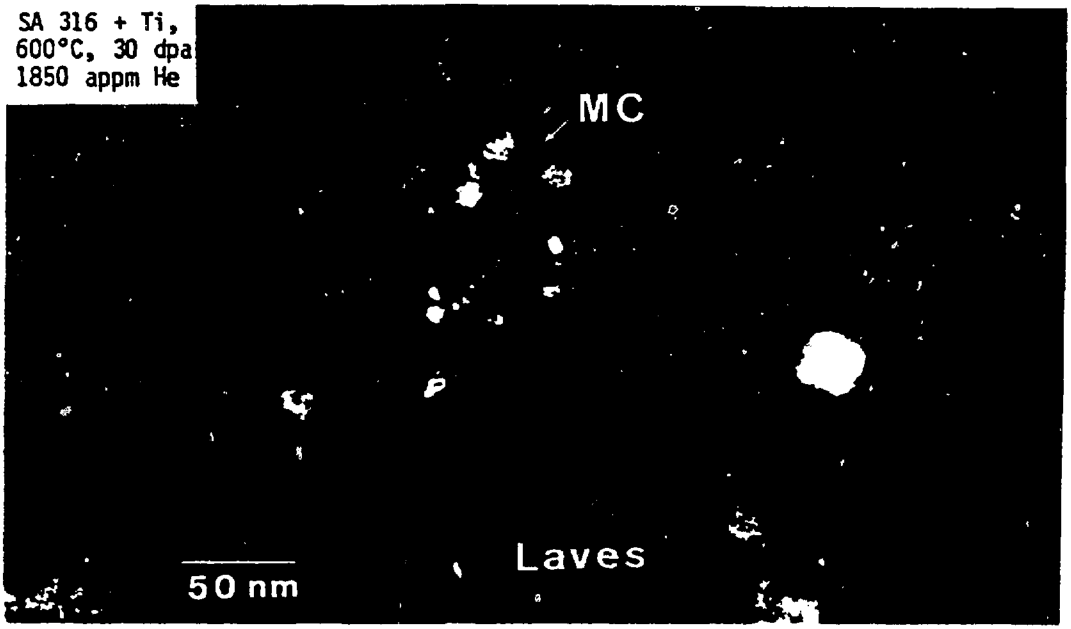
II. MICROSTRUCTURAL DEVELOPMENT IN PATH A PCA

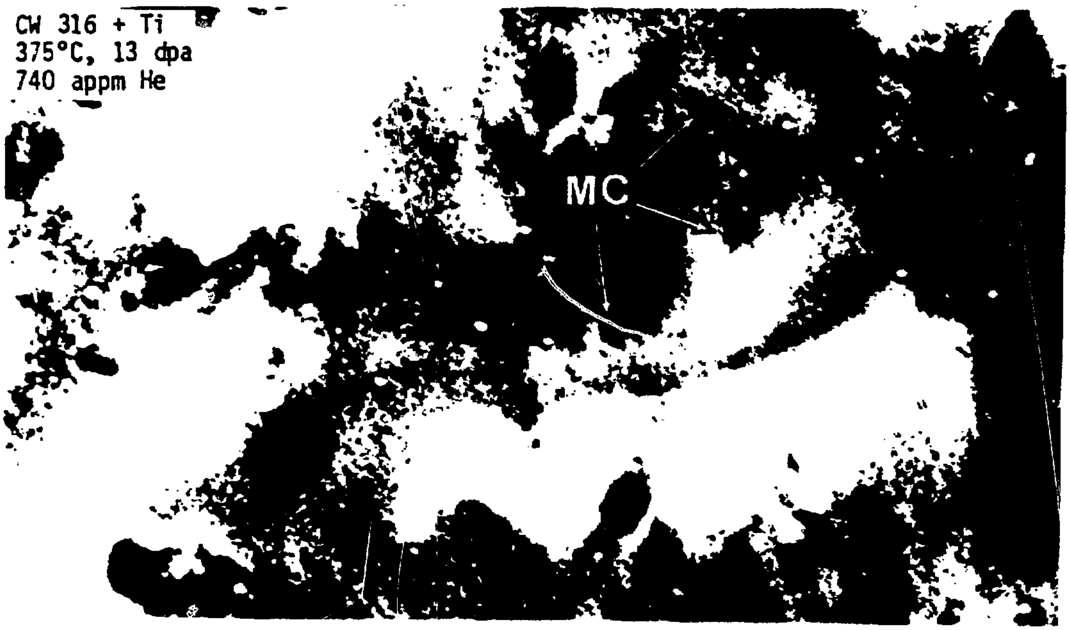
COMBINED BOTH TEM AND PHYSICAL METALLURGY FOR:

- SOLVING FABRICATION PROBLEMS
  (1) INITIAL INHOMOGENEITY OF MATERIAL
  (2) NITRIDE FORMATION
  (3) GRAIN SIZE CONTROL
- INVESTIGATING TIME-TEMPERATURE-PRECIPITATION (TTP) OF 0, 10, AND 25% CW MATERIAL
- BLENDING THERMAL-MECHANICAL TREATMENTS FOR COMPLEX MICROSTRUCTURES WITH PROPER FABRICATION TO PRODUCE SPECIMEN MATERIAL

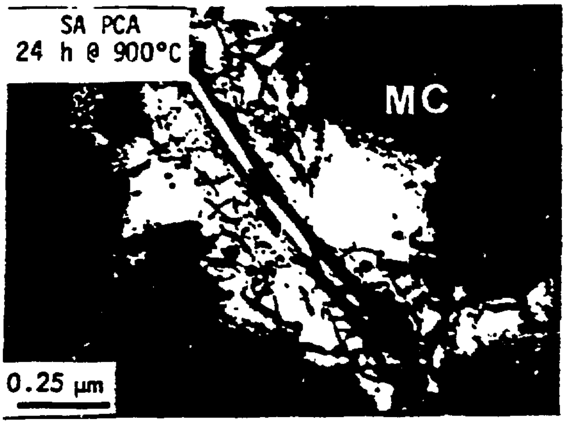
BASICS OF THE HELIUM-MC TRAPPING EFFECT

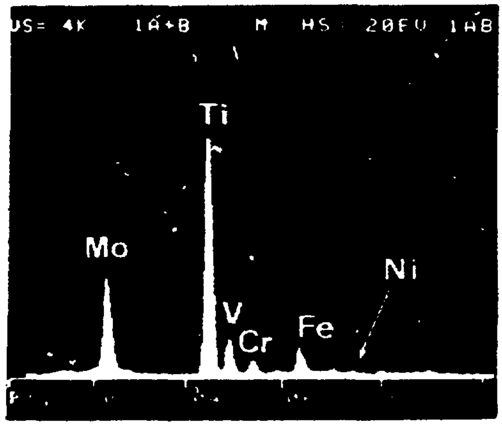
- EXCLUSIVE TRAPPING OF HELIUM AT MC-PARTICLE INTERFACES
- EFFECTIVE TRAPPING OF THE HELIUM IN MANY SMALL BUBBLES
- MC FORMATION IS ENHANCED DURING IRRADIATION WITHOUT SIGNIFICANT Ni OR Si ENRICHMENT
- MC BEHAVES AS A VACANCY BIASED SINK DUE TO LARGE OVERSIZE MISFIT





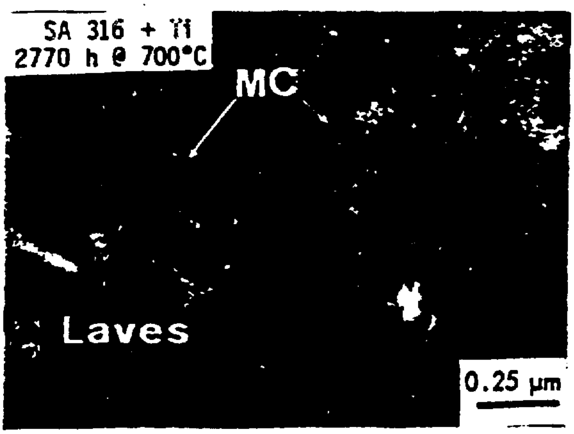
MICROSTRUCTURES DEVELOPED BY THERMAL AGING ARE RELEVANT
BECAUSE MC COMPOSITION IS NOT AFFECTED MUCH BY IRRADIATION

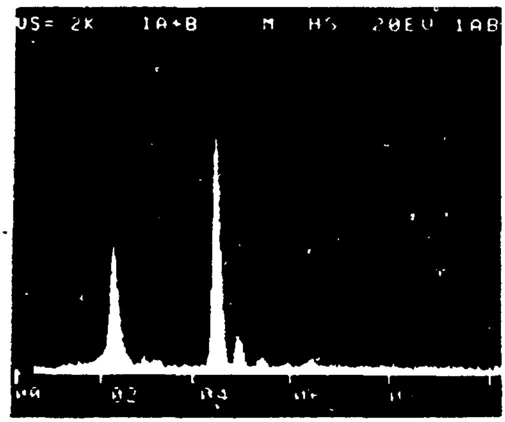




Content (wt %)

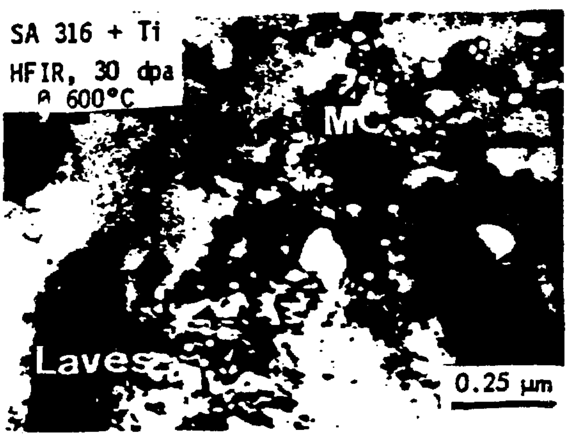
Si — nd
Mo — 33.0
Ti — 51.0
V — 3.5
Cr — 3.5
Mn — nd
Fe — 8.0
Ni — 1.0

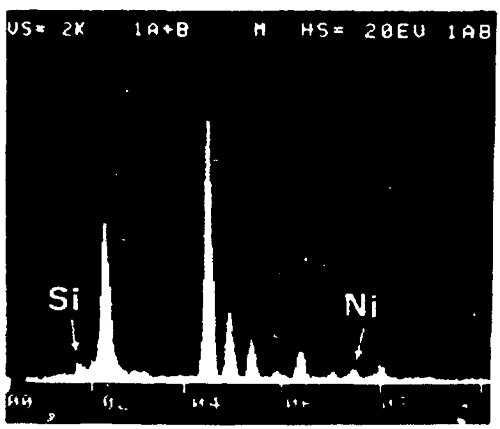




Content (wt %)

Si — nd
Mo — 48.0
Ti — 45.5
V — 2.0
Cr — 2.0
Mn — nd
Fe — 2.0
Ni — 0.5



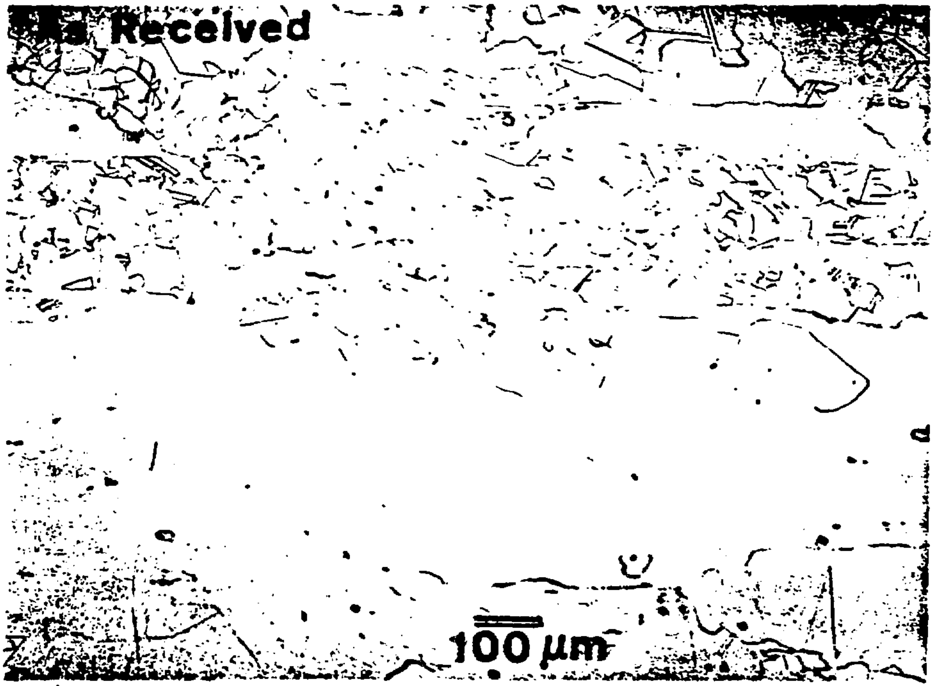


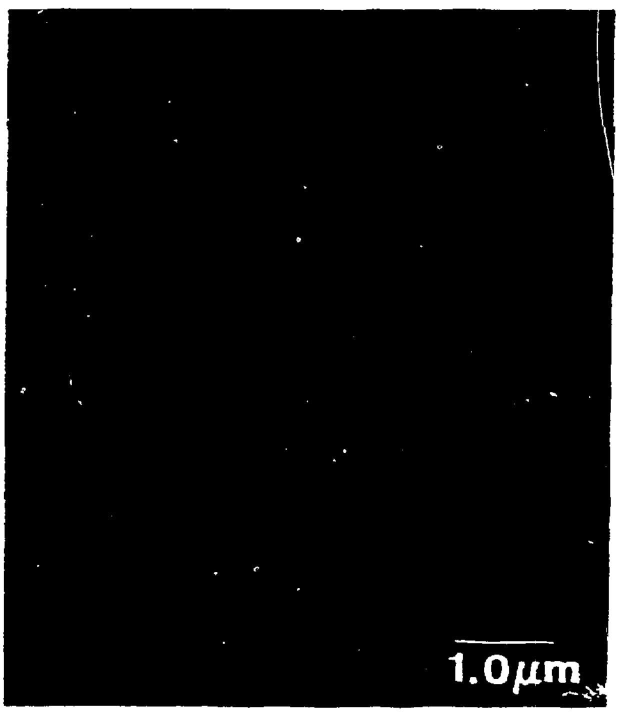
Content (wt %)

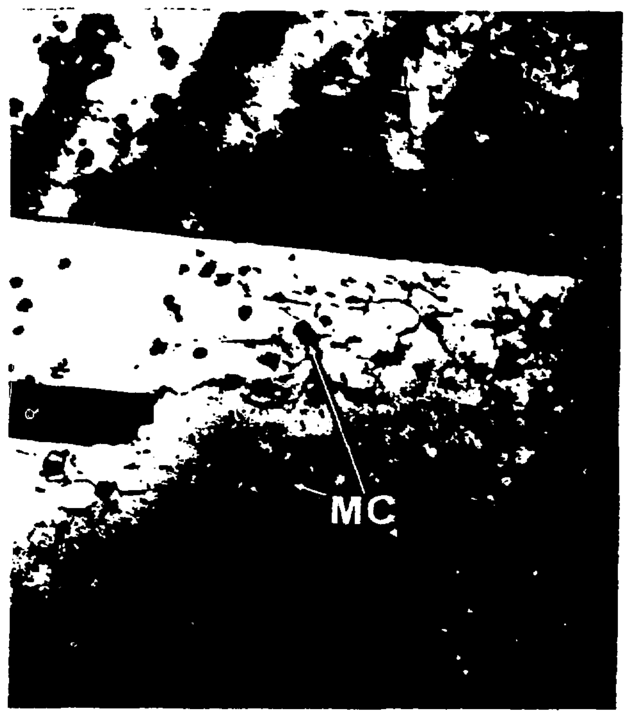
Si — 1.0
Mo — 41.0
Ti — 37.5
V — 6.5
Cr — 5.0
Mn — 0.5
Fe — 5.5
Ni — 3.0

FABRICATION PROBLEMS

INITIAL PATH A PCA MATERIAL HAD GROSSLY INHOMOGENEOUS,
STRINGERED MC DISTRIBUTION







A SYSTEMATIC STUDY OF FABRICATION VARIABLES ON MICROSTRUCTURE WAS INITIATED. TEM AND METALLOGRAPHY WERE INCLUDED AT ALMOST EVERY STEP.

RESULTS

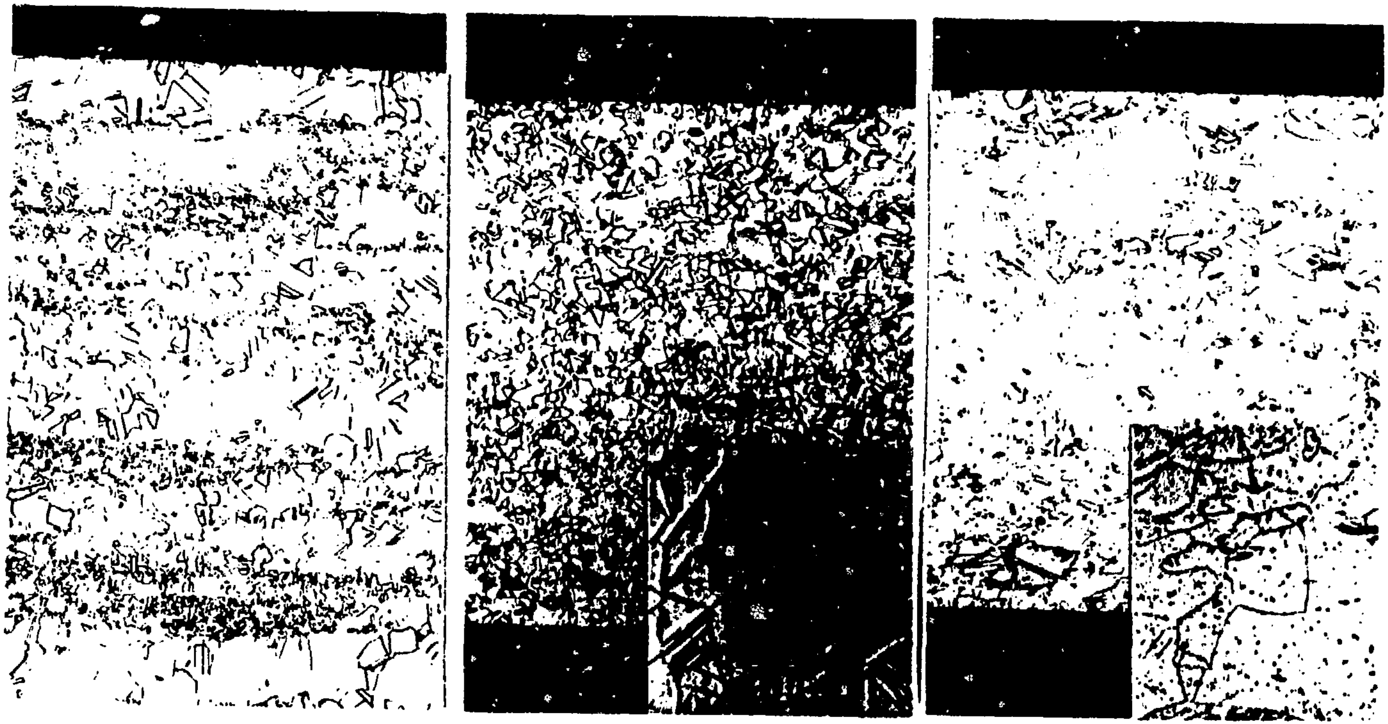
- HOMOGENIZATION AT 1200 TO 1275°C FOR 24 h COULD ELIMINATE THE ORIGINAL PROBLEM

- SOLUTION ANNEALING OR RECRYSTALLIZATION THAT PRECIPITATED MC FOLLOWED BY COLD WORKING WAS A DISASTER, PRODUCING STRINGERS

- PROPER CARE TO MAINTAIN HOMOGENEITY DURING FABRICATION WAS VERY IMPORTANT FOR PRODUCING GOOD SPECIMEN MATERIAL

HIGH-TEMPERATURE HEAT TREATING (~ 1175°C) TO PRESERVE HOMOGENEITY ALSO LEADS TO VERY COARSE GRAIN SIZE

BUT TREATMENTS (LIKE 1 h at 1050°C) OR SLOW HEATING RATE TO PRODUCE MC CAN RESULT IN NONUNIFORM GRAIN SIZE



STRINGERS AFTER RECRYSTALLIZATION AT 1050°C AND COLD WORKING

SA AT 1050°C SLOW HEATING RATE

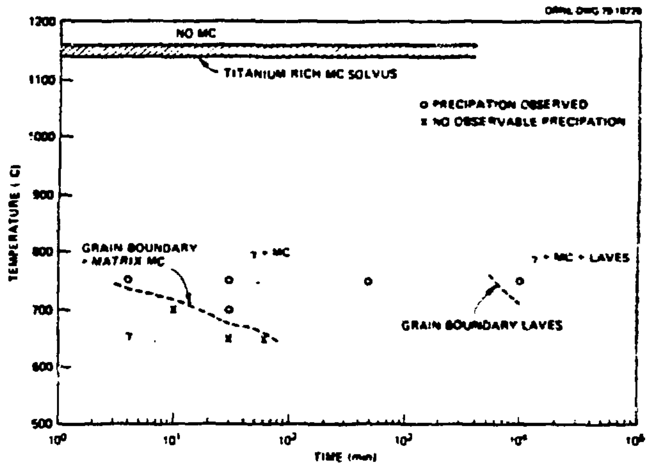
SA AT 1050°C FAST HEATING RATE

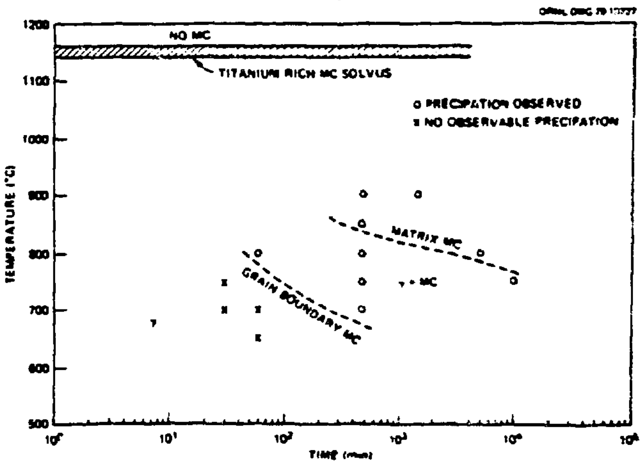
100 μm

TTP CURVES FOR PATH A PCA

THESE SHOW:

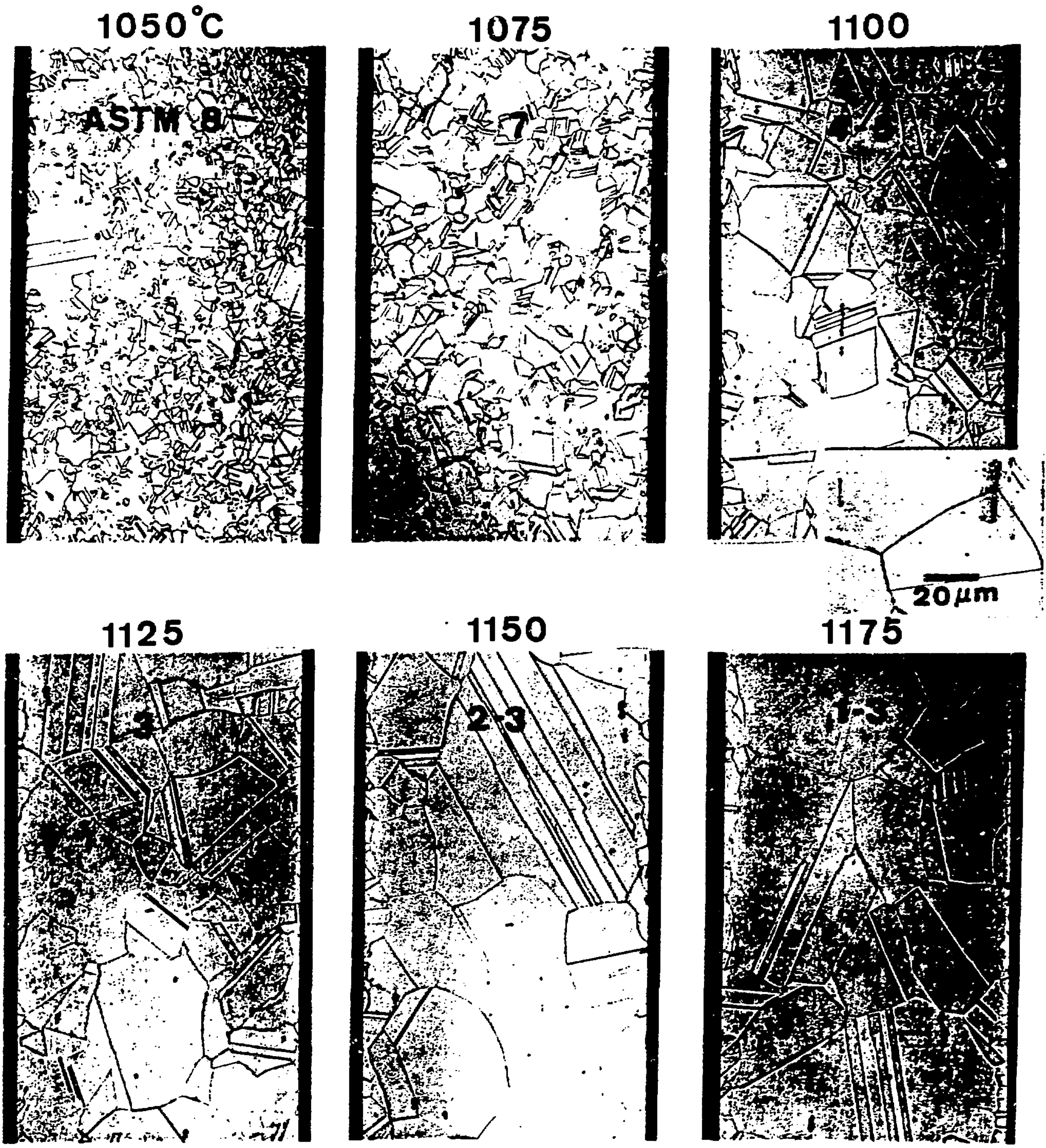
- MC PRECIPITATION IS VERY FAST IN CW MATERIAL (5 min OR LESS AT 750 TO 800°C)
- MC PRECIPITATION IS QUITE SLOW IN SA MATERIAL (SEVERAL HOURS AT 800 TO 900°C)
- IT SHOULD BE POSSIBLE TO RECRYSTALLIZE CW MATERIAL PRIOR TO PRECIPITATION
  - FAST HEATING RATE
  - HIGH POWER AND CONTINUOUS HEATING





IF MC DOES NOT FORM, GRAIN SIZE IS A FUNCTION

OF ANNEALING TEMPERATURE



30 min.

TTP CURVE INVESTIGATION ALSO YIELDS

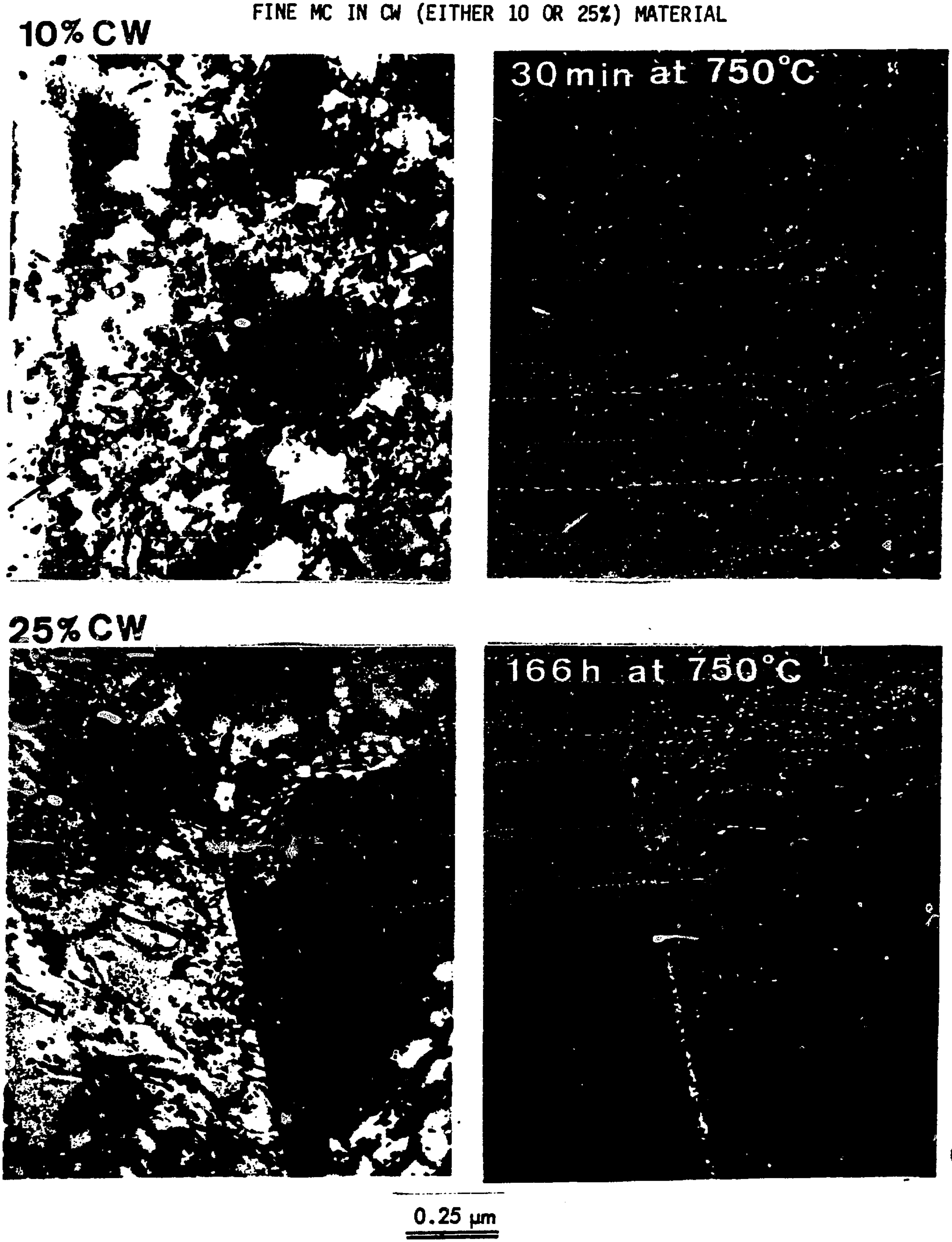
- MICROSTRUCTURES AVAILABLE IN PCA
- THERMAL MECHANICAL TREATMENTS TO PRODUCE THEM

SEVERAL REASONS WHY PCA IS A GOOD ALLOY:

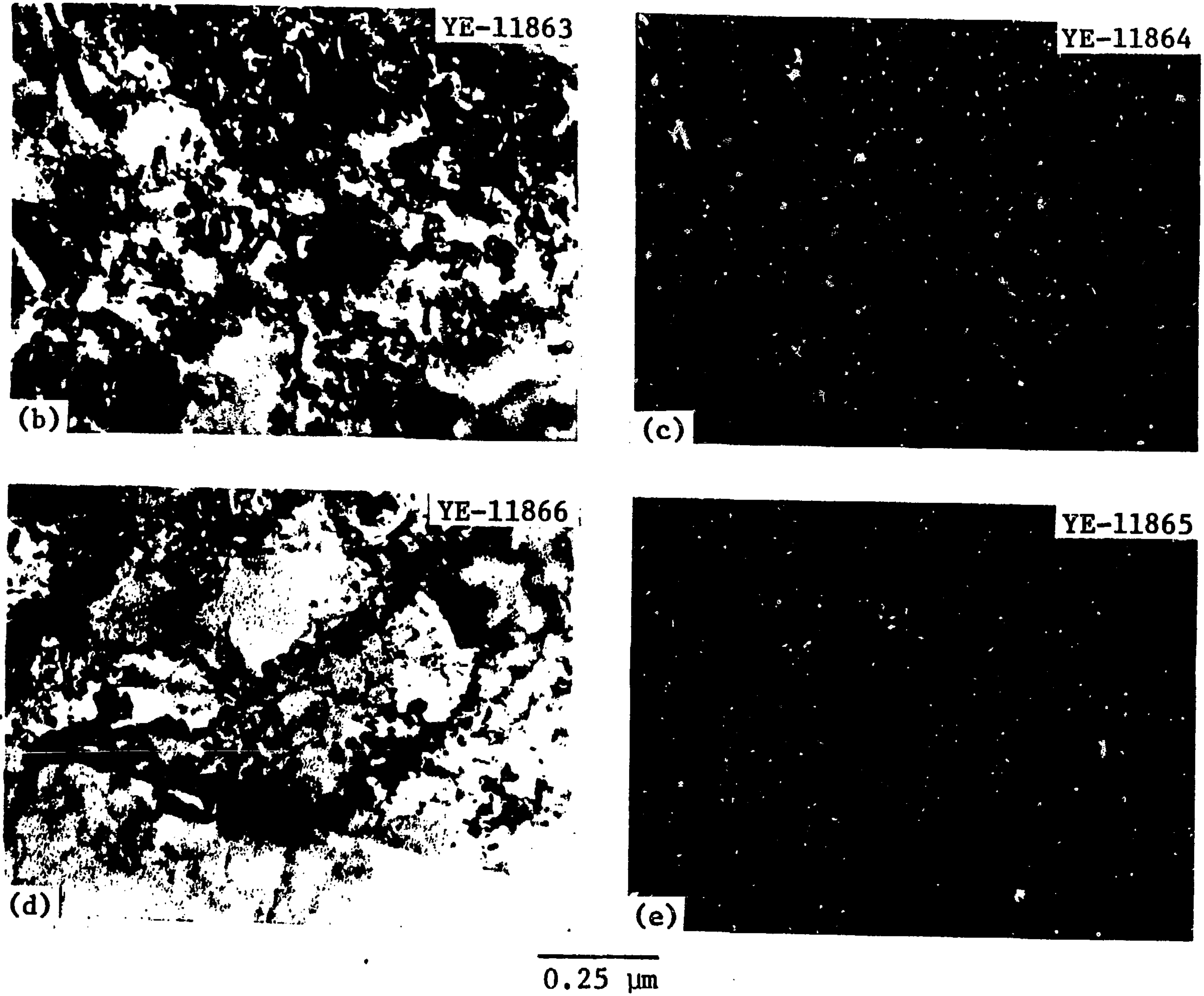
- MC FORMS AT GRAIN BOUNDARIES AS WELL AS AT MATRIX
- BOTH GRAIN BOUNDARY AND MATRIX DISTRIBUTIONS OF MC ARE VARIABLE
- ETA AND $M_{23}C_6$ ARE NOT OBSERVED AND ONLY A SMALL AMOUNT OF LAVES PHASE


8h at 800°C
COARSE MC IN SA MATERIAL
24h at 900°C
0.25 μm
1 μm
15min at 1175°C
8 h at 800°C
8 h at 900°C

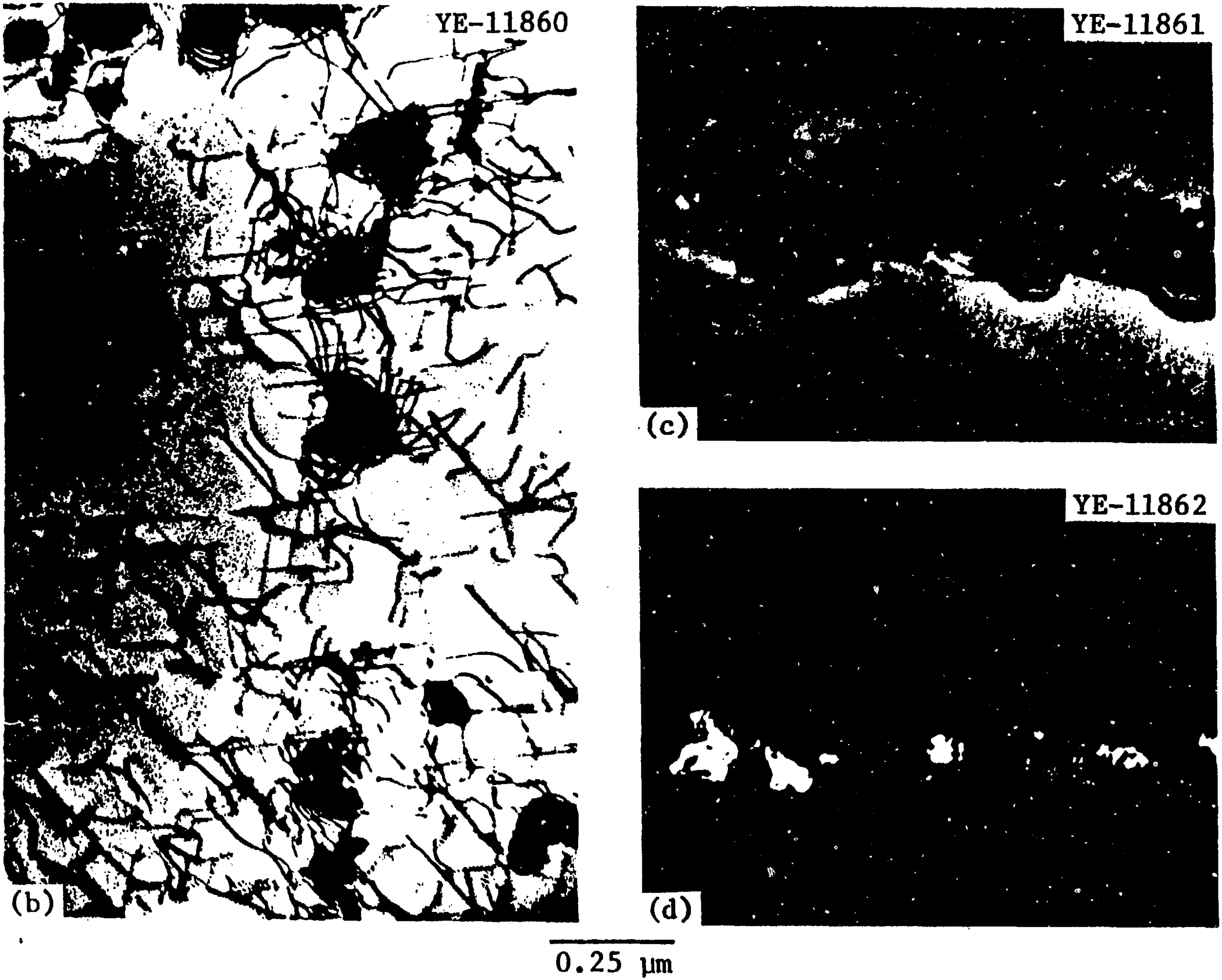
FINE MC IN CW (EITHER 10 OR 25%) MATERIAL



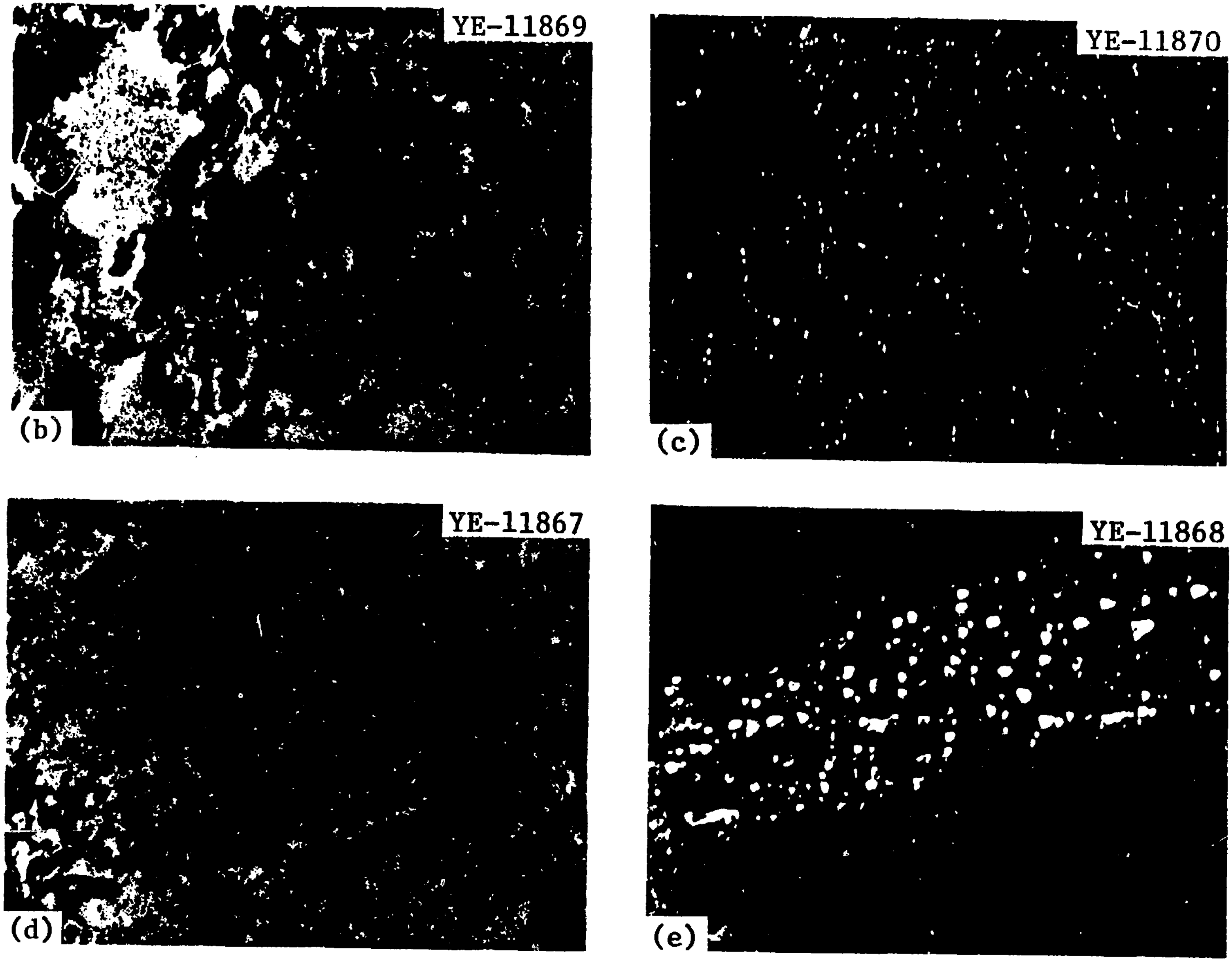
Microstructure C
CW 25% + 2h at 750°C



Microstructure B1
SA + 8h at 800°C + gh at 900°C



icrostructure B2
+ 8H at 800°C + 25% CW + 2h at 750°C



0.25 μm

SUMMARY

- ALLOY DEVELOPMENT HAS INCLUDED:
  - MICROSTRUCTURAL DESIGN
  - MICROSTRUCTURAL CHARACTERIZATION
  - FABRICATION STUDIES
- RESULTS
  - WE ARE ABLE TO FABRICATE HOMOGENEOUS MATERIAL THAT HAS COMPLETELY VARIABLE GRAIN SIZE
  - WE HAVE FOUND THERMAL MECHANICAL TREATMENTS CAPABLE OF VARYING GRAIN BOUNDARY OR MATRIX MC DISTRIBUTIONS OVER A WIDE RANGE
  - WE CAN COMBINE THE TWO TO PRODUCE VARIABLE AND INNOVATIVE PREIRRADIATION MICROSTRUCTURES IN A VARIETY OF FINAL SPECIMEN TYPES

THIS PROGRAM OFFERS BALANCE. IT FINDS AND INITIATES STUDIES OF BASIC PRINCIPLES AND THEN APPLIES THESE WITH GOOD PHYSICAL METALLURGY FOR SUCCESSFULLY